



Intra-laboratory quality control for chicken raw materials

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ABSTRACT. In recent times, food and water laboratories have aimed to improve their processes to meet market demands. In order to contribute to the efficacy and development of these processes, new methods must be sought, among which is intra-laboratory quality control. This research study portrays the use of this method in a food and water laboratory, in order to analyze the intra-laboratory statistical control of chicken raw materials. To that end, the methodology applied was: Exploratory data analysis; Cochran's test, to assess the homogeneity of variances; Dixon's Test, to verify whether the means are part of the same distribution; calculation of repeatability and reproducibility; calculation of the uncertainty of the mean; Snedecor's F method, to compare the variances; and Dixon's test, to compare laboratory variances. The analysis parameter analyzed in the studied raw material was the proficiency of technicians with regard to the accuracy of analyses of total coliform count in the raw materials prior to chilling. The final results showed low data variation around the mean, as well as significant reliability of the obtained results.

Keywords: food health, intra-laboratory analysis, proficiency.

Controle de qualidade intralaboratorial na matéria-prima da carcaça de frango de corte

RESUMO. Nos últimos tempos, os laboratórios de análises de alimentos e água vêm buscando melhorar seus processos, visando atender à expressiva demanda de mercado e para contribuir com a eficiência e o desenvolvimento desses processos busca novos métodos, dentre eles o controle de qualidade intralaboratorial. O presente estudo mostra a utilização desse método em um laboratório de análises de alimentos e água com objetivo de analisar o Controle Estatístico Intralaboratorial, da matéria-prima na carcaça de frango. Para tanto, foram definidos a metodologia e os métodos estatísticos a serem analisados tais como: análise exploratória de dados - Teste de Cochran para verificar a homogeneidade das variâncias; Teste Dixon para verificar se as médias fazem parte de uma mesma distribuição; Cálculo da Repetibilidade e Reprodutibilidade, Cálculo da incerteza da média, Método F - Snedecor para comparar as variâncias; e Teste Dixon para comparação das Variâncias do Laboratório. O Parâmetro de análise monitorado na matéria-prima estudada foi a eficiência dos técnicos quanto à precisão das análises para contagem de Coliformes Totais, nas carcaças de frangos antes do processo de resfriamento. Os resultados finais demonstraram pequena variação dos dados em torno da média e significativa confiabilidade dos resultados obtidos.

Palavras-chave: segurança do alimento, análises intralaboratorial, proficiência.

Introduction

Laboratory Best Practices are part of the integrated quality management system used to assess organizational procedures and the conditions under which laboratory research studies are planned, performed, tested, monitored, recorded, inspected and archived. The objective of intra-laboratory quality control is to meet the requirements of rule NBR ISO 17025:2005 and the demands of Brazil's Ministry of Agriculture, Livestock and Supply – MAPA.

According to studies proposed by Henry (1999), Quality Control for Food and Water Laboratories

are systems devised to recognize and minimize lab analysis errors; they provide analysts with the criteria to identify and analyze laboratory performance, in order to obtain reliable and safe results.

The same author also reports that quality control programs in clinical laboratories were introduced in Brazil during the 1970s-1980s through the National Quality Program (PNQ) of the Brazilian Society of Clinical Analysis (SBAC).

Quality control may be defined as every systematic action to support and maintain laboratory services in order to meet client needs. It is regarded as a component of the Quality Management System (MOTTA et al., 2001).

The view of statistical control of the process is associated with lower variability and provides improved quality levels in production results. This becomes important when dealing with best processes – meaning not only better quality, but lower costs as well. Costs decrease for two reasons, namely: sampling inspection and waste reduction (PALADINI, 2004).

Intra-laboratory trials are assays performed by several technicians on a standard sample, in order to evaluate proficiency and obtain the grouped repeatability variance value of the laboratory for each assay technique (LOPES, 2003).

Based on the above considerations, it can be observed that there currently is great concern by food and water laboratories regarding the accuracy of the results obtained in routine laboratory analysis procedures. In that regard, statistical techniques meet that need. Intra-laboratory quality control emerges as a prospect for better evaluating and demonstrating the reliability of the obtained results.

The objective of this work was to perform an intra-laboratory statistical control study on chicken raw materials at a food and water laboratory, in order to assess the efficiency and accuracy of the obtained results.

Material and methods

The methodology used in this study was quantitative and descriptive. The following statistical methods were used to analyze the research in question:

1. Exploratory data analysis (Cochran's test) (LOPES, 2003), to assess the homogeneity of variances;

2. Dixon's test (LOPES, 2003), to verify whether the means are part of the same distribution;

3. Calculation of repeatability and reproducibility (LOPES, 2003);

4. Calculation of the uncertainty of the mean, using the formula:

$$U\bar{X} = t(N-1; 0,025) \times \frac{S_{Total}}{\sqrt{N}} \quad (1)$$

In which:

$U\bar{X}$ = uncertainty of the mean;

S_t = total standard deviation;

n = total number of determinations;

$t(n-1; 0.025)$ = Student's t variable.

5. Snedecor's F method to compare the variances;

6. Dixon's test to compare Laboratory variances.

Using Cochran's test, the homoscedasticity of variances is compared; that is, it is possible to assess

whether the variance of the obtained results by a group is excessive compared to the other groups; it is a one-sided test. The C test is performed according to the following formula:

$$C = \frac{S^2_{max}}{\sum S^2_i} \quad (2)$$

S^2_{max} : is the maximum variance of the group.

$\sum S^2_i$: is the sum of all sample variances in the group.

The decision rule is as follows: if the calculated value of C is lower than the obtained C value (LOPES, 2003), the variances are equal to 5% significance.

Dixon's test compares the individual values obtained by a single operator or the mean values obtained by several operators, or yet differences between two results obtained by several operators; it is a two-sided test (LOPES, 2003).

$$Q_{Calculated} = \frac{Z(H) - Z(H-1)}{Z(H) - Z(1)} \quad (3)$$

In which $Z(H)$, $Z(H-1)$ and $Z(1)$ are minimum and maximum order statistics, respectively.

Repeatability refers to tests performed under conditions as constant as possible, known as repeatability conditions. The results of mutually independent tests are obtained using the same assay method, identical materials, by the laboratory, same operator, and using the same equipment over short intervals. The standard deviation of the test result obtained under repeatability conditions is known as repeatability standard deviation. It is a parameter of the dispersion of the distribution of test results. With the repeatability standard deviation, the so-called repeatability r value is calculated: the modulus of the difference between the two test results obtained under repeatability conditions is then calculated. The likelihood that this difference is lower than the repeatability r value is 95% (LOPES, 2003).

$$r = tx\sqrt{2}xSr \quad (4)$$

In which t is the value found in Student's t -table, with $\alpha = 0.05$ (two-sided) and with the residual degree of freedom:

S_r : is the repeatability standard deviation.

Reproducibility refers to tests performed under varying conditions, named reproducibility conditions. The results are obtained using the same assay method and identical materials, but in different laboratories, by different operators and using different equipment, with large time intervals between tests. The standard deviation of the test result obtained under reproducibility conditions is known as reproducibility standard deviation. It is a parameter of the dispersion of the distribution of test results. Com o reproducibility standard deviation, the so-called reproducibility R value is calculated: the modulus of the difference between the two test results obtained under reproducibility conditions is then calculated. The likelihood that this difference is lower than the reproducibility R value is 95% (LOPES, 2003).

$$R = tx\sqrt{2}xSR \quad (5)$$

In which t is the value found in Student's t -table, with $\alpha = 0.05$ (two-sided) and with the degree of freedom

SR: is the reproducibility standard deviation.

There are several tests involving the parameters of a multiple regression model or multiple correlation coefficient. The standard test is the analysis of variance, in which the explained variation is compared with the unexplained variation of the dependent variable. This relationship features Snedecor's F distribution with k and $(n-k-1)$ degrees of freedom, with k equal to the number of regressors and n equal to sample size. The calculated statistical parameter $F_{\text{calculated}}$ is then compared to table parameter $F_{(k,n-k-1)}$. If $F_{\text{calculated}} > F_{(k,n-k-1)}$, the null hypothesis of non-existence of a linear relationship is rejected, according to the indications of α % significance (1% or 5%) of evaluation norm (in order to reach Degree III); in other words, the regression equation is accepted. For the obtained model, that test is performed using the $F_{\text{calculated}}$ equation, calculated by the formula:

$$F_{\text{Calculated}} = \frac{SQM_{\text{TECHNICIANS}}}{SQM_{\text{RESIDUALS}}} \quad (6)$$

In which:

SQM: Sum of the Squares of Technicians;

SQM: Sum of the Squares of Residuals.

Results and discussion

In order to apply the intra-laboratory techniques in the food and water laboratory used in the study, it

was verified that the laboratory has adopted a complete management system for laboratory best practices in all procedures, from the moment samples are received to the process of issuing analysis reports.

Table 1 shows a summary of the results for mean and variance per technician.

In Table 1 are explained the results of the analyses obtained by each technician, the mean of results, standard deviation and variance. From those data, it can be observed in the obtained results that the variation was greater for technicians B and C, compared to the mean and variance of the other technicians. This indicates there was greater variability, which may be related to different factors inherent to the performed analysis. As such, certain aspects can be taken into account, such as: skill and experience of each technician, the manner in which the procedure was interpreted, and the condition of the sample at the time of analysis. Other factors that can favor the variability of a given process or procedure have to do with the environment, calibration and accuracy of equipment, among others.

Cochran's test was performed to assess whether the variances are equal (homoscedasticity) in each technician. The obtained result demonstrates that $C_{\text{calculated}} = 0,328$ was lower than $C_{\text{Fixed}} = (4,4,5\%) = 0,684$. Thus, the variances have 5% de significance, as follows:

$$C_{\text{calculated}} = \frac{S^2_{\text{máx}}}{\Sigma S^2_i} = \frac{2,92 \times 10^8}{8,38 \times 10^8} = 0.348$$

and

$$C_{\text{Fixed}} = (4,4,5\%) = 0,684.$$

Thus: as $C_{\text{Calculated}} < C_{\text{Fixed}}$, the variances have 5% significance.

Table 1 also includes the summary of means; it can be observed that the distribution of the means is in ascending order, considering that the highest mean is for technicians C and D.

Thus, the statistical calculation using Dixon's method to verify whether the means are part of the same distribution:

$$Q_{\text{Calculated}} = \frac{Z(H) - Z(H-1)}{Z(H) - Z(1)} = \frac{1,22 \times 10^5 - 1,20 \times 10^5}{1,22 \times 10^5 - 1,043 \times 10^5} = 0.1129$$

$$\text{with } Q_{\text{Fixed}}(4, 5\%) = 0,829$$

with $Q_{\text{Calculated}} < Q_{\text{Fixed}}$ at 5% significance, the means belong to the same distribution.

normality of a process, calibration and gauging of equipment. Quality management techniques are modern concepts that aim to contribute efficiently to the survival of companies in ever-changing markets (CIMA; URIBE-OPAZO, 2010).

Conclusion

The study performed herein demonstrated what technician proficiency with regard to the accuracy of analyses is within a statistically efficient normality. The analyses of variance were higher for technicians B and C, and the results of technicians C and D showed the greatest homogeneity – that is, lowest data dispersion – featuring a higher representativeness of the mean, as their coefficients of variation were near $CV = 20\%$. In an overall evaluation, a small variation of the data was detected around the mean, as well as significant representativeness of the obtained results.

In that sense, the study also showed that the application of statistical techniques proved efficient in a context of continuous improvement, considering that the use of the scientific method is important in decision-making. Therefore, it was demonstrated that the use of information technology through statistical techniques is of fundamental importance for the process of integrated laboratory management.

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Received on March 1, 2011.

Accepted on September 12, 2011.

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